



# The solution treatment of *in-situ* sub-micron TiB<sub>2</sub>/2024 Al composite

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## ABSTRACT

In this work, we studied the solution treatment of *in-situ* TiB<sub>2</sub>/2024 Al composite. In the composite, TiB<sub>2</sub> particles have ranged from 30 to 500 nm, and the second phases (SPs) of  $\theta$  and S were identified to coexist with TiB<sub>2</sub>. Furthermore, mechanical properties subjected to different solution temperatures (STs) for T4 state were investigated. It was found that the increase of ST can effectively improve mechanical properties of composite. The optimum ST for the composite was 505 °C, which was higher than ST (500 °C) typically used in 2024 Al alloy. To understand this phenomenon, a modified diffusion-controlled dissolution model for the composite has been proposed. In the classical model for the alloy, the dissolution process should depend on the initial radius of SP, solution time and ST. However, the TiB<sub>2</sub> aggregates around SPs should act as particle shells to reduce the effective contact area between the alloy matrix and SPs. This covering effect has hindered the dissolution of SPs in the composite. In consideration of this effect, a modified diffusion-controlled dissolution model was proposed for the composite. The increase of the ST that was more approaching to the eutectic temperature was a required strategy to enhance the SP dissolution.

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## 1. Introduction

The Al-Cu-Mg alloy is one of the most commonly used aerospace Al alloys, which has been intensively developed recently in order to meet the future industry requirement. The particulate reinforced metal matrix composite is one of the developed materials with higher mechanical properties in comparison with the metallic counterpart. For example, the particulate reinforcements like SiC [1–4], B<sub>4</sub>C [5,6], and Al<sub>2</sub>O<sub>3</sub> [7,8] particles have been widely investigated in the Al-Cu-Mg alloy composites. In these composites, the particles are usually added by *ex-situ* methods with the larger particle size in micron scale and higher particle volume fractions. The SiC<sub>p</sub>/Al composites are the typical materials in this category. For instance, Angers et al. [2] reported that the yield strength of the 2024 Al alloy (T6) was increased from 402 MPa to 595 MPa by the addition of SiC particles (~2–6 μm) from 0 vol.% to 25.3 vol.%, but the elongation was sharply decreased from 10% to 2.4%. Varma et al. [3] found that the strength and elongation has decreased greatly with the increasing sizes of SiC particles in the SiC<sub>p</sub>/(Al-Cu-Mg) composites. As the size of SiC particles was increasing from 1.4 μm to 62.8 μm, the elongation has decreased from 11.7% to 1.9%. These results showed that it was detrimental to the overall ductility in the cases of the high particle volume fractions and large sizes of particle reinforcements.

Recently, the *in-situ* TiB<sub>2</sub>/Al alloy composites have been successfully prepared via the salt-metal reaction route, which showed the better and balanced mechanical properties [9–13]. Nevertheless, TiB<sub>2</sub> particles are prone to coexist with the second phases (SPs) during solidification process. The aggregation of TiB<sub>2</sub> particles is related to the impingement caused by the high concentration of neighboring particles preventing rotation to a more energetically favorable crystallographic orientation during solidification [14,15]. For the Al-Cu-Mg alloy composites, the solution treatment is one of the important methods to optimize the mechanical properties. Allowing the SPs of  $\theta$  (CuAl<sub>2</sub>) and S (Al<sub>2</sub>CuMg) phases to dissolve into the matrix during solution treatment can enhance the yield strength by solution strengthening or cluster hardening [16–19]. The solution temperatures for the 2024 Al alloy [20–22] and 2024 Al composites [2,17–19] are usually chosen below 505 °C. However, the solution may be greatly impeded by reinforced particles during solution treatment since the particles have coexisted with the SPs. In this case, the solution treatment on these composites should not be treated the same like the matrix alloys. A large amount of undissolved SPs in composites will lead to the reduction of mechanical properties if they cannot be solubilized adequately. Nevertheless, the little attention has been given to the effect of the solution treatment on such composites.

In this paper, the *in-situ* TiB<sub>2</sub>/2024 Al composite were synthesized via the salt-metal reaction technique. The microstructural characterization found the SPs have a trend to coexist with TiB<sub>2</sub> particles in the composite. Based on this microstructural characteristic, a modified diffusion-controlled dissolution model was proposed for the composite to understand the reason for the increasing solution temperatures required in the composite.

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**2. Experimental procedures**

The 2024 Al alloy and 5 wt.% TiB<sub>2</sub>/2024 Al composite were used in this study. The 2024 matrix alloy has a nominal composition (in wt.%) of Al-4.3Cu-1.7Mg-0.5Mn. The *in-situ* TiB<sub>2</sub>/2024 Al composite was fabricated by the salt-metal reaction method. The pre-mixed K<sub>2</sub>TiF<sub>6</sub> and KBF<sub>4</sub> salts were added to the molten alloy. Then, the melt was stirred using a blade paddle mixer for 20 min. Hereby, the slag was skimmed out from the melt surface. Finally, the melt was cast into a permanent mold. The as-cast 2024 alloy and TiB<sub>2</sub>/2024 Al composite ingots were homogenized at 490 °C for 24 h, and then were extruded at 450 °C with the extrusion ratio of 35: 1. The solution treatment was performed using a Nabertherm furnace, subsequently following by water quenching. Afterwards, the natural aging (T4) treatment was carried out on the alloy and composite for four days at least. The differential scanning calorimetry (DSC) investigations were run on a DSC 204F1 apparatus at a rate of 10 °C/min. Microstructures and tensile fracture surfaces were examined by scanning electron microscopy (SEM, NOVA NanoSEM 230) equipped with energy dispersive spectroscopy (EDS). The mechanical properties were evaluated by the tensile test, which was carried out on the Zwick/roell Z200 testing machine at a strain rate of 10<sup>-3</sup> s<sup>-1</sup> according to the ASTM E8/E8M-15a standard [23].

**3. Results**

*3.1. Microstructure*

Fig. 1 shows backscattered electron (BSE) micrographs of the as-extruded 2024 Al alloy and *in-situ* 5 wt.% TiB<sub>2</sub>/2024 Al composite before the solution treatment. The banded structures are observed, with the grains being elongated along the extruded direction in both samples. It is clear that the second phases (SPs) of θ, S and/or θ + S eutectics in the alloy are relatively more uniform and smaller than those in the composite. In the composite, TiB<sub>2</sub> particles bands are observed closely

aggregated with SPs near grain boundaries in Fig. 1(b), while TiB<sub>2</sub> particles distribute uniformly inner the grain bands. The different flow rates of the matrix and TiB<sub>2</sub> particles during the hot extrusion have led to the TiB<sub>2</sub> particles band structures in the composite.

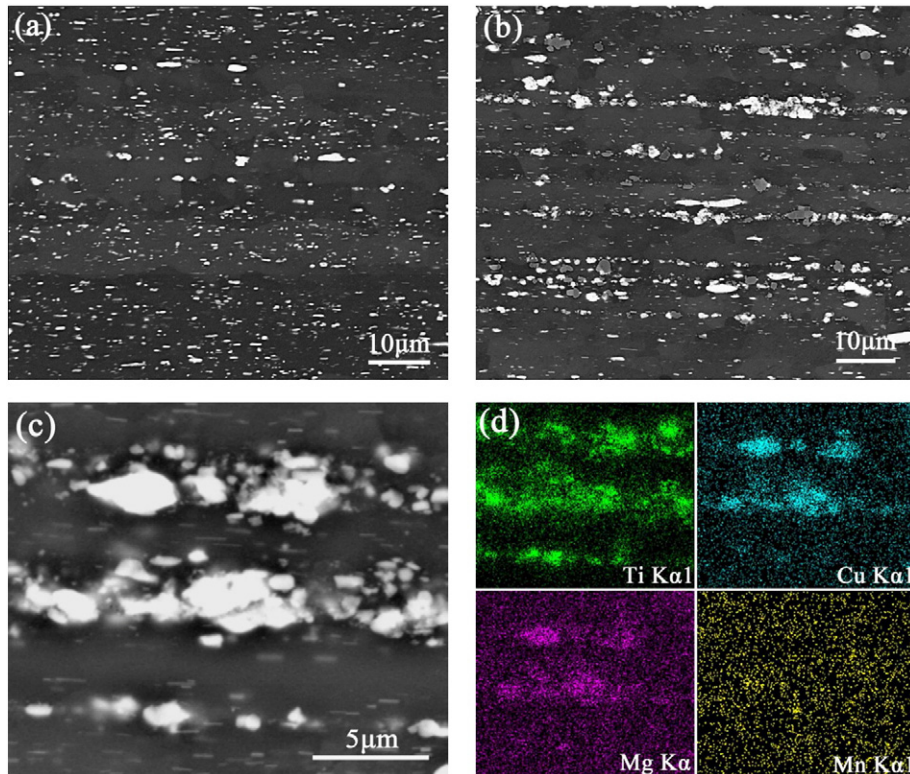
The element mapping analyses of the region of TiB<sub>2</sub> particle bands are shown in Fig. 1(c) and (d). It reveals that the SPs rich in Cu and Mg elements have coexisted with TiB<sub>2</sub> particles, which indicates the solution treatment will be strongly influenced by the TiB<sub>2</sub> particle bands. Besides, the Mn element is distributed in the uniform manner, signifying the T phases (Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub>) that has mainly precipitated during the homogenization is uniformly distributed in the composite.

Fig. 2(a) shows a higher magnification view of the typical morphology of TiB<sub>2</sub> particles located at the grain boundaries. Although TiB<sub>2</sub> particles are in large volume fraction near grain boundaries, they are actually uniform within the micron scale. In Fig. 2(b), the size of the TiB<sub>2</sub> particles has laid in the range of 30–500 nm. It is accepted that ceramic particles in the large micron size are easily broken during extruding [3,24–26], which can introduce voids or micro-cracks in materials. However, the sub-micron particles of TiB<sub>2</sub> are so small that their integrity can be kept in the composite.

*3.2. Tensile property*

Fig. 3 presents the dependences of mechanical properties of the 2024 alloy and TiB<sub>2</sub>/2024 Al composite on the different solution time. The solution temperature for both samples is initially chosen at a conventional temperature of 500 °C which is typically used for 2024 Al alloy. It is obvious that the strength of the composite is much higher than that of the alloy. The strengthening mechanisms have been widely investigated in former literatures [10,25,27].

Before 1 h, the yield strength (σ<sub>0.2</sub>) and the elongation (δ) of both alloy and composite have an obvious increasing trend. The σ<sub>0.2</sub> peaks of the alloy and composite are reached almost at the same time



**Fig. 1.** BSE micrographs of as-extruded (a) 2024 Al alloy and (b) *in-situ* (5 wt.%) TiB<sub>2</sub>/2024 Al composite before solution treatment, (c) a higher magnification BSE micrograph of the *in-situ* TiB<sub>2</sub>/2024 Al composite in the TiB<sub>2</sub> particles bands regions, and (d) corresponding EDS element mapping analyses.

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